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POLYPROPYLENE FIBER WITH HIGH HEAT RESISTANCE

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### Abstract

#### Purpose

The purpose of the present invention is to produce a polypropylene fiber or yarn with high heat resistance suitable for use as a cement reinforcement material that does not undergo fusion at autoclave curing temperatures in the range of 175-180°C and that has superior shape retention.

#### Means to solve

In a polypropylene fiber or yarn with high heat resistance produced by hot-melt molding a homopolypropylene resin having an isotactic pentad fraction in the range of 96-98.5% and melt-flow rate (230°C, 2.16 kg load) in the range of 0.1-30 g/10 min and drawing, a polypropylene fiber or yarn with high heat resistance characterized by the fact that the heat shrinkage factor at 170°C for 10 min is 10% or below and the fusion peak temperature is 178°C or above.

### Claim

1. A polypropylene fiber or yarn with high heat resistance produced by hot-melt molding a homopolypropylene resin having an isotactic pentad fraction in the range of 96-98.5% and melt-flow rate (230°C, 2.16 kg load) in the range of 0.1-30 g/10 min and drawing, which polypropylene fiber or yarn with high heat resistance is characterized by the fact that the heat shrinkage factor at 170°C for 10 min is 10% or below and the fusion peak temperature is 178°C or above.

2. The polypropylene fiber or yarn with high heat resistance described in Claim 1, characterized by the fact that a heat treatment is provided for the polypropylene fiber or yarn

with high heat resistance after drawing at a temperature in the range of 170-195°C under restraint tension.

3. The polypropylene fiber or yarn with high heat resistance described in Claim 1 or 2, characterized by the fact that the fusion peak of the polypropylene fiber or yarn with high heat resistance before the heat treatment with restraint measured under restraint tension exists at 180°C or above.

4. A cement reinforcement fiber made of the polypropylene fiber or yarn with high heat resistance described in one of Claims 1-3.

#### Detailed explanation of the invention

[0001]

##### Technical field of the invention

The present invention pertains to a polypropylene fiber or yarn with high heat resistance and the invention further pertains to a polypropylene fiber or yarn with high heat resistance used for cement reinforcement fiber.

[0002]

##### Prior art

Historically, cement moldings are used for building materials such as interiors, exteriors, and roofing. In the past, products containing asbestos fibers have been widely used as reinforcement materials for cement moldings, but with recent environmental problems, the adverse effect of asbestos on health is gaining attention and use of replacement fibers is increasing each year. For reinforcement fibers to replace the above-mentioned asbestos, polyolefin fibers, in particular, polypropylene fibers and polyethylene fibers can be mentioned, and their use is on the increase as replacement fibers that can be used without adverse effects on the human body.

[0003]

Cement requires a curing process during molding. Curing is achieved in an autoclave (10 kgf/cm<sup>2</sup>) for several tens of hours at a temperature in the range of 170-180°C. However, the fusion point of standard polypropylene fibers is in the range of 160-165°C; thus, fusion takes place during the aforementioned curing and the polypropylene fails to exist in the cement concrete after curing. When the curing temperature for the cement concrete is reduced to 165-170°C, for example, a long curing time is required and productivity is reduced.

[0004]

Problems to be solved by the invention

Based on the above background, the purpose of the present invention is to produce a polypropylene fiber or yarn with high heat resistance suitable for use as a cement reinforcement material that does not undergo fusion in an autoclave at a curing temperature of 175-180°C and that has superior shape retention.

[0005]

Means to solve the problems

As a result of much research conducted by the present inventors in an effort to eliminate the above-mentioned existing problems, the researchers discovered that it was possible to control the thermal shrinkage factor under high temperature to a low value and to increase the fusion peak temperature when a heat treatment is provided for a homopolypropylene resin having specific stereoregularity and flow properties at a specific temperature under restraint tension after hot-melt drawing, and as a result, the present invention was accomplished.

[0006]

In other words, the first invention of the present invention is a polypropylene fiber or yarn with high heat resistance characterized by the fact that the heat shrinkage factor at 170°C for 10 min is 10% or below and the fusion peak temperature is 178°C or above in a polypropylene fiber or yarn with high heat resistance produced by hot-melt molding a homopolypropylene resin having an isotactic pentad fraction in the range of 96-98.5% and melt-flow rate (230°C, 2.16 kg load) in the range of 0.1-30 g/10 min and drawing.

[0007]

Furthermore, the second invention of the present invention is the polypropylene fiber or yarn with high heat resistance described in the first invention characterized by the fact that a heat-treatment is provided for the polypropylene fiber or yarn with high heat resistance after drawing at a temperature in the range of 170-195°C under restraint tension.

[0008]

Furthermore, the third invention of the present invention is the polypropylene fiber or yarn with high heat resistance described in the first or second invention characterized by the fact that the fusion peak of the polypropylene fiber or yarn with high heat resistance before the heat-treatment with restraint measured under restraint tension exists at 180°C or above.

[0009]

Furthermore, the fourth invention of the present invention is a cement reinforcement fiber made of the polypropylene fiber or yarn with high heat resistance described in the first to the third inventions.

[0010]

Embodiment of the invention

In the following, the present invention is explained in further detail.

#### 1. Polypropylene resin

The polypropylene resin used in the present invention is a homopolypropylene resin having an isotactic pentad fraction (hereinafter referred to as IPF), which is the index of stereoregularity, of 96-98.5% and satisfies the melt-flow rate (hereinafter referred to as MFR) of 0.1-30 g/10 min, preferably, 0.5-25 g/10 min at 230°C and under a load of 2.16 kg. When the IPF is 96% or below, the fusion temperature of the homopolypropylene itself is reduced, and it is not suitable to be used as a cement reinforcement material; on the other hand, when the IPF is 98.5% or above, spinning stability such as stability of the thread is lost as a result of an excessively high rate of crystallization of the molten resin at the time of hot-melt spinning. Furthermore, when the MFR is 0.1 g/10 min or below, an increase in the pressure at the die exit occurs at the time of hot-melt molding (mainly at the time of spinning of the fiber); on the other hand, when the MFR is 30 g/10 min or above, the polymer component is reduced in the polypropylene resin, oriented crystals in the fiber or yarn are reduced after drawing, and as a result, the fusion temperature of the fiber or yarn is reduced, and the material is not suitable to be used as a cement reinforcement material. It is further desirable when the molecular weight distribution ( $M_w/M_n$ ) of the homopolypropylene is in the range of 3.5-12, and 4-9 is further desirable.

[0011]

For the homopolypropylene resin used in the present invention, conventional modifiers for polyolefins may be used in combination according to the intended application. For example, antioxidants, ultraviolet absorbers, light stabilizers, crystalline nucleating agents, organic carboxylic acids, antistatic agents, surfactants, neutralizing agents, dispersants, epoxy stabilizers, plasticizers, lubricants, antibacterial agents, flame retardants, fillers, blowing agents, foaming co-agents, crosslinking agents, crosslinking co-agents, pigments, etc., can be mentioned. For antioxidants, phenolic antioxidants, phosphoric antioxidants, sulfur type antioxidants, amine type antioxidants, vitamins, etc., can be mentioned. For neutralizing agents that double as dispersants,

metal soaps, hydrotalcites, lithium aluminum composite hydroxylate, silicates, metal oxides, metal hydroxides, etc., can be mentioned. Furthermore, it is effective to add 0.1-20 parts by weight of a hydrophilic polymer such as polyethylene glycol and polyethylene oxide so as to increase dispersibility of the fiber or yarn in the cement.

[0012]

2. Method for production of polypropylene fiber or yarn with high heat resistance

The polypropylene fiber or yarn with high heat resistance of the present invention is produced by hot-melt molding the above-mentioned homopolypropylene resin to form an amorphous fiber, drawing to form a fiber or yarn, and providing a heat treatment under restraint tension.

[0013]

In general, an amorphous fiber is produced by the hot-melt molding method using a pellet type or powder type homopolypropylene resin raw material. For example, a multifilament hot-melt spinning device or monofilament hot-melt spinning device is used to produce an amorphous yarn. Furthermore, when the material is cut after passing through the flat die or ring die, a tape for drawing (split yarn) is produced. Subsequently, drawing is done with a drawing machine.

[0014]

Drawing is achieved in a single stage or in a multistage device consisting of two or more stages. The drawing temperature is in the range of 70-150°C and the drawing operation is done using an oven, a hot plate, hot drawing roll, infrared, hot water (wet heat), etc., as a heat source. The drawing ratio is in the range of 1.5-10 times, preferably, 2-7 times, for fibers, and 2-20 times, preferably, 4-18 times, for a yarn. When drawing is done in a multistage device, the temperature can be increased in a stepwise fashion and final drawing is done at a temperature in the range of 160-195°C. In this case, the heat treatment described below may be omitted. In other words, when the drawing roll temperature at the time of drawing is set to a temperature in the range of 160-190°C, an inline heat treatment under restraint tension is made possible.

[0015]

It is desirable when the drawn polypropylene fiber or yarn has a fusion peak temperature measured under a restraint state of 180°C or above. In other words, when the fusion peak measured under restraint tension of the drawn polypropylene fiber or yarn before the heat-treatment described below, in specific terms, when the fusion peak measured at a scanning

rate of 10°C/min as the fiber is wound around a metal such as an aluminum sheet to prevent shrinkage of the fiber and placed on a DSC measurement plate, is 180°C or above, production of a fiber or yarn having an adequate resistance to the heat treatment described below is possible. When the aforementioned fusion peak is 180°C or below, fusion of the fiber or yarn occurs at the time of heat treatment under high temperature and under restraint tension, which is not desirable.

[0016]

A heat treatment is provided for the polypropylene fiber or yarn with high heat resistance drawn as described above under restraint tension at a temperature of 170-195°C. In general, a heat-treatment is provided at a temperature in the range of 120-160°C, preferably in the range of 130-150°C in the past, but crystallization at the oriented crystal part is likely to be promoted when a heat treatment is provided for the drawn fiber under restraint tension in the present invention, and an increase in the fusion point is made possible, and fusion of the drawn fiber or yarn does not occur at a temperature above the fusion point, and production of a fiber with a low shrinkage factor and high fusion point is made possible.

[0017]

In other words, when a heat treatment is applied at a heat-treatment temperature in the range of 170-195°C, preferably 175-190°C, for 2-60 min, preferably 5-40 min, it is possible to achieve a heat shrinkage factor of the polypropylene fiber of 10% or below at 170°C for 10 min, preferably 10% or below at 175°C for 10 min. Furthermore, it is possible to shift the fusion peak start-up temperature, fusion peak temperature, and fusion end temperature at DSC measurement toward the high temperature side, and it is possible to achieve a fusion peak temperature of 178°C or above. When the heat-treatment temperature is 170°C or below, the fusion peak temperature of the fiber or yarn with a low drawing ratio is capable of achieving approximately 173°C, at most, and when the temperature exceeds 195°C, fusion of fiber or yarn takes place and heat treatment is difficult.

[0018]

### 3. Cement reinforcement fiber and reinforced cement molding

As described above, the heat shrinkage factor is low in the polypropylene fiber or yarn with high heat resistance of the present invention under high temperature and the fusion point is shifted toward the high-temperature side and is suitable to be used as a cement reinforcement fiber. Especially when curing is done in an autoclave used for concrete at a temperature of 175-180°C, the shape is retained and it can be used as effectively as a reinforcement material.



[0019]

For the cement for which the polypropylene fiber or yarn with high heat resistance of the present invention is used as a cement reinforcement material, for example, hydraulic setting cements such as portland cement, white portland cement, aluminous cement, silica cement, magnesia cement, and pozzuolana cement, air-hardening cements such as gypsum and coal, special cements such as acid-resistant cement, etc., can be mentioned.

[0020]

Furthermore, production of a cement composition that utilizes the above-mentioned cement is made possible when an inorganic material such as calcium carbonate and magnesium hydroxide, optional aggregates such as pebbles and sand, thermosetting water-soluble resins such as pulp, paraffin, wax and resol type phenolic resin, a variety of polymer emulsions, curing accelerators, curing inhibitors, water reducing agents, etc., are added to one or more types of the above-mentioned cements. Upon curing of the above-mentioned cement composition, the mixing ratio of the cement and water, when water is added to the cement composition, that is, the C/W ratio, is preferably in the range of 1-10. When the C/W ratio is below 1, the amount of water becomes too high and an adequate strength cannot be achieved upon curing of the cement; on the other hand, when said ratio exceeds 10, the flow properties of the cement composition become inferior.

[0021]

When the polypropylene fiber or yarn with high heat resistance of the present invention is used as a cement reinforcement material, the application form varies depending on the shape of the fiber or yarn. For example, when a yarn is used as the cement reinforcement material, a method where the yarn is fastened to the partially cured cement composition using a lock bolt, etc., and the cement composition is supplied is used. Furthermore, when the fiber is used as a cement reinforcement material, preferably, the fiber is cut to a length of 3-30 mm and mixed in the above-mentioned cement composition. In this case, uniform dispersing of the fiber in the cement composition is less likely to occur when the length of the fiber exceeds 30 mm; on the other hand, an adequate reinforcement effect cannot be achieved at 3 mm or below.

[0022]

Furthermore, in general, the amount of cement reinforcement material used is in the range of 0.1-30 parts by weight for 100 parts by weight of the cement composition, and in the range of 0.5-15 parts by weight is further desirable. When the amount of the cement reinforcement material included is 0.1 part by weight or below, an adequate reinforcement effect

cannot be achieved; on the other hand, when the amount exceeds 30 parts by weight, uniform dispersing of the cement reinforcement material is less likely to be achieved.

[0023]

Furthermore, upon mixing of the polypropylene fiber or yarn with high heat resistance with the cement composition as a cement reinforcement material, it is desirable when a treatment is provided with a surfactant, etc., to increase the affinity with the cement.

[0024]

A variety of cement products can be mentioned as fiber-reinforced cement moldings that utilize the polypropylene fiber or yarn with high heat resistance of the present invention. For example, underwater structures such as tetrapods, railway structures such as bridges and tunnels, structures such as buildings, houses (interiors and exteriors), wall surfaces, revetments, roof tiles, etc., can be mentioned.

[0025]

#### Application examples

The present invention is explained in further detail below, but the present invention is not limited to the application examples below. Furthermore, test methods used in the present invention are as shown below.

[0026]

#### (1) IPF

The isotactic factor by pentad unit in the polypropylene molecular chain is measured by a nuclear magnetic resonance spectral analysis ( $^{13}\text{C}$ -NMR) based on isotopic carbon according to the method reported in *Macromolecules*, Vol. 6, p. 925 (1973) by A. Zambelli et al. In other words, the isotactic pentad factor is the propylene unit factor where five continuous propylene monomer units are isotactically bonded. Wherein, assignment of the peak is done according to the corrected version of the aforementioned document described in *Macromolecules*, Vol. 8, p. 687 (1975). In specific terms, measurement of the isotactic pentad unit is done based on the intensity factor of the mmmm peak in the total absorption peak in the methyl carbon region of the  $^{13}\text{C}$ -NMR spectra.

[0027]

(2) MFR

According to the specification of JIS K 7210, measurement was done under a load of 2.16 kg and at 230°C.

(3) Mw/Mn (molecular weight distribution)

GPC was used for the measurement.

[0028]

(4) Fusion peak temperature under a restraint state

Approximately 4 mg of a sample fiber or yarn were wrapped around an aluminum sheet and an arrangement was made to prevent shrinkage of the fiber at the time of temperature increase, and the fusion peak temperature was measured at a scanning rate of 10°C/min starting from room temperature.

[0029]

(5) Fusion peak start-up temperature, fusion peak temperature, and fusion end temperature

Approximately 10 mg of drawn sample fiber or yarn were used and the measurement was made by DSC (Differential Scanning Calorimetry) at a scanning rate of 10°C/min starting from room temperature. In this case, the fusion peak start-up temperature is the nodal point of contact of the base line and fusion peak start-up line.

[0030]

(6) Heat shrinkage factor

The polypropylene fiber or yarn with high heat resistance was retained in an oven heated to 170°C and 175°C for 10 min and the shrinkage ratio is defined as the heat shrinkage factor.

[0031]

(7) Fiber shape retention after curing in an autoclave

After curing in an autoclave, the concrete test piece was cracked and an evaluation was made based on the shape of the fiber cross section based on the criteria shown below.

O : complete shape of the fiber is left behind in the cross section.

Δ: fiber is partially dissolved at the cross section and a change of shape is observed.

X: fiber is dissolved and a change of shape is observed in the cross section.

[0032]

Application Example 1

For the homopolypropylene powder with an IPF of 97.2%, MFR of 1.5 g/10 min and molecular weight distribution of 4.5, 0.05 part by weight each of tetrakis[methylene-3-(3,5-di-t-butyl-4-hydroxyphenyl)propionate]]methane and tris(2,4-di-t-butylphenyl)phosphite (both products of Chiba Special Chemicals Co.) were added as antioxidants, then, 0.05 part by weight calcium stearate was added as a neutralizing agent, and blending was done in a supermixer, and hot-melt mixing was done by an extruder with a diameter of 50 mm $\phi$  at a temperature of 230°C and screw rotation of 75 rpm to produce polypropylene pellets.

[0033]

Hot-melt spinning was done for the aforementioned pellets by a multifilament spinner (die: 0.8 mm $\phi$  x 30 holes) equipped with a gear pump at a spinning temperature of 280°C and take-up speed of 300 m/min to produce an amorphous yarn of approximately 20 denier. Furthermore, drawing was done at a feed rate of 50 m/min, feed roll temperature of 90°C, heater temperature at a drawing point of 130°C and drawing roll temperature of 160°C to produce a yarn drawn by 3.7 times at a maximum drawing ratio of 4.0 times. The DSC chart obtained by measuring the fusion peak temperature under a restraint state of the drawn yarn is shown in Figure 1. The fusion peak temperature was 201°C.

[0034]

Both ends of the drawn yarn were fastened to prevent shrinkage by heat and it was placed in a gear oven heated to 180°C for 30 min and a heat treatment was applied to produce a heat-resistant polypropylene fiber. Measurements were made of the fusion peak start-up temperature, fusion peak temperature, fusion end temperature, and heat shrinkage factor of the fiber produced. The results obtained are shown in Table 1 below. Furthermore, the DSC chart obtained upon measurement of fusion peak temperature, etc., is shown in Figure 2.

[0035]

In order to increase affinity with the cement, a polyoxyalkylene glycol type surfactant (trade name: Leocon [transliteration] 1015B, product of Lion Co.) was coated onto the drawn fiber produced as described above after the heat treatment (0.1 wt% for the fiber); then, the fiber was cut to form lengths of 15 mm, and added to a cement composition comprising a normal portland cement (product of Taiheiyo Cement Co.), No. 8 quartz sand, pulp, and water at a weight ratio of normal portland cement : No. 8 quartz sand : pulp : water = 100:100:3:60, and stirring and mixing were provided by an Omni mixer. In this case, the mixing ratio of the cement

composition and the above-mentioned fiber-like cement reinforcement material was cement composition: fiber-like cement reinforcement material = 100:1 at a volume ratio.

[0036]

The cement and fiber-like cement reinforcement material mixture produced as described above was poured into a mold form with a length of 80 mm, width of 30 mm, and height of 20 mm and then atmospheric pressure steam curing was provided for one day, and curing in an autoclave was provided for one day. The concrete test piece was cracked and the shape of the fiber was examined. The results obtained are shown in Table 1 below.

[0037]

Atmospheric pressure steam curing: Curing was done for 2-5 h at 23°C and the temperature was increased to 65°C at a rate of 20°C/h, and isothermal curing was done for 3-5 h. Subsequently, the temperature was reduced to 23°C in 10-15 h and slow cooling was achieved.

[0038]

Curing in an autoclave: The mold was removed and the test piece was placed in an autoclave pot, and heated to 160°C and pressurized to 10 atm in 3 h and the isothermal temperature and pressure were retained for 3 h. Subsequently, the temperature was increased to 180°C in 1 h and 10 atm was retained; then, water was poured into the space between the pot and the outer wall and cooling was done over 7-10 h.

[0039]

As shown in Table 1 and Figure 2, the heat shrinkage factor at 170°C for 10 min is 0% and the fusion peak start-up temperature, fusion peak temperature, and fusion end temperature of the fiber-like cement reinforcement material based on DSC measurement were 175°C, 179°C, and 184°C, respectively, and in comparison to the prior art, a significant increase in the fusion point is observed, and furthermore, good shape retention after autoclave curing at 180°C was confirmed.

[0040]

#### Application Example 2

The heat-treatment temperature was changed to 183°C and production of a fiber-like cement reinforcement material with a drawing ratio of 3.7 times was achieved and a test sample was produced as in Application Example 1. The results obtained are shown in Table 1 and Figure

2. As a result, the heat shrinkage factor at 170°C for 10 min is 0% and the fusion peak start-up temperature, fusion peak temperature, and fusion end temperature of the fiber-like cement reinforcement material based on DSC measurement were 177°C, 182°C, and 187°C, respectively, and a significant increase in the fusion point is observed, and furthermore, good shape retention after autoclave curing at 180°C was confirmed.

[0041]

#### Application Example 3

A homopolypropylene powder with an IPF of 96.8%, MFR of 0.5 g/10 min, molecular weight distribution of 4.7 was used and production of a fiber-like cement reinforcement material having a drawing ratio of 3.2 was achieved and a test sample was produced as in Application Example 1. The results obtained are shown in Table 1 and Figure 2. As a result, the heat shrinkage factor at 170°C for 10 min is 0% and the fusion peak start-up temperature, fusion peak temperature, and fusion end temperature of the fiber-like cement reinforcement material based on DSC measurement were 176°C, 181°C, and 185°C, respectively, and a significant increase in the fusion point is observed, and furthermore, good shape retention after autoclave curing at 180°C was confirmed.

[0042]

#### Application Example 4

For a homopolypropylene powder with an IPF of 97.0%, MFR of 2 g/10 min and molecular weight distribution of 4.5, 0.05 part by weight each of tetrakis[methylene-3-(3,5-di-t-butyl-4-hydroxyphenyl)propionate]methane and tris(2,4-di-t-butylphenyl)phosphite (both products of Chiba Special Chemicals Co.) was added as antioxidants; then, 0.05 part by weight calcium stearate was added as a neutralizing agent, and then, 0.04 part by weight of 2,5-dimethyl-2,5-di(t-butylperoxy)hexane (Perhexa [transliteration] 25B, product of Japan Fats and Oils Co.) was added and blending was done by a supermixer, and hot-melt mixing was done by an extruder with a diameter of 50 mmφ at a temperature of 230°C and screw rotation of 75 rpm to produce a polypropylene pellet with an MFR of 10 g/10 min and molecular weight distribution of 3.7.

[0043]

Hot-melt spinning was conducted for the pellets produced by a multifilament spinner (die: 0.8 mmφ x 30 holes) equipped with a gear pump at a spinning temperature of 280°C and take-up speed of 300 m/min to produce an amorphous yarn of approximately 20 denier. Furthermore, drawing was done at a feed rate of 50 m/min, feed roll temperature of 90°C, heater

temperature at the drawing point of 130°C and drawing roll temperature of 160°C to produce a yarn drawn to 4.5 times. The DSC chart obtained by measuring the fusion peak temperature under the restraint state of the drawn yarn is shown in Figure 1. The fusion peak temperature was 183°C.

[0044]

Both ends of the drawn yarn were fastened to prevent shrinkage by heat and placed in a gear oven heated to 180°C for 30 min and heat treatment was applied to produce a heat-resistant polypropylene fiber. Measurements were made for the fusion peak start-up temperature, fusion peak temperature, fusion end temperature, and heat shrinkage factor of the fiber produced. The results obtained are shown in Table 1 and Figure 2.

[0045]

The heat-resistant polypropylene fiber cement reinforcement material was used and the state after curing in an autoclave as in the case of Application Example 1 was examined. As a result, the heat shrinkage factor at 170°C for 10 min is 0% and the fusion peak start-up temperature, fusion peak temperature, and fusion end temperature of the fiber-like cement reinforcement material based on DSC measurement were 174°C, 179.5°C, and 185°C, respectively, and a significant increase in the fusion point is observed, and furthermore, good shape retention after autoclave curing at 180°C was confirmed.

[0046]

#### Comparative Example 1

The drawing roll temperature at the time of drawing was changed to 110°C and the heat treatment was omitted and production of a fiber-like cement reinforcement material with a drawing ratio of 3.7 times was achieved as in Application Example 1, and measurement of each property was done as in Application Example 1 and the state after curing in an autoclave was examined as described above. The results obtained are shown in Table 1 and Figure 2. As a result, the heat shrinkage factor at 170°C for 10 min was 77% and the fusion peak start-up temperature, fusion peak temperature, and fusion end temperature of the fiber-like cement reinforcement material based on DSC measurement were low values of 158°C, 165°C and 173°C, respectively, and good shape retention after autoclave curing at 180°C was absent.

[0047]

Comparative Example 2

The drawing roll temperature at the time of drawing was changed to 110°C and the heat-treatment temperature was changed to 165°C and a fiber-like cement reinforcement material with a drawing ratio of 3.7 times was produced as in Application Example 1, and measurement of each property was done as in Application Example 1 and the state after curing in an autoclave was examined as described above. The results obtained are shown in Table 1 and Figure 2. As a result, the heat shrinkage factor at 170°C for 10 min was 65% and the fusion peak start-up temperature, fusion peak temperature, and fusion end temperature of the fiber-like cement reinforcement material based on DSC measurement were low values of 167°C, 169°C and 177°C, respectively, and good shape retention after autoclave curing at 180°C was absent.

[0048]

Comparative Example 3

A homopolypropylene powder with an IPF of 94.2%, MFR of 2 g/10 min and molecular weight distribution of 5.2 was used and the drawing roll temperature was changed to 110°C and the heat-treatment temperature was changed to 155°C and a fiber-like cement reinforcement material with a drawing ratio of 4.0 was produced as in Application Example 1, and measurement of each property was done as in Application Example 1 and the state after curing in an autoclave was examined as described above. The results obtained are shown in Table 1. As a result, measurement of the heat shrinkage factor at 170°C for 10 min was not possible due to fusion, and the fusion peak start-up temperature, fusion peak temperature, and fusion end temperature of the fiber-like cement reinforcement material based on DSC measurement were low values of 158°C, 163°C and 168°C, respectively, and good shape retention after autoclave curing at 180°C was absent.

[0049]

Comparative Example 4

In Comparative Example 3, when the heat-treatment temperature of 170°C was used, fusion of the fiber took place during the course of the heat treatment and production of a fiber-like cement reinforcement material was not possible.

[0050]

Comparative Example 5

A homopolypropylene powder with an IPF of 96.7%, MFR of 40 g/10 min and molecular weight distribution of 4.2 was used and spinning temperature of 250°C, drawing roll temperature



of 110°C, and heat-treatment temperature of 160°C were used and a fiber-like cement reinforcement material with a drawing ratio of 5.0 times was produced as in Application Example 1, and measurement of each property was done as in Application Example 1 and the state after curing in an autoclave was examined as described above. The results obtained are shown in Table 1. As a result, measurement of the heat shrinkage factor at 170°C for 10 min was not possible due to fusion, and fusion peak start-up temperature, fusion peak temperature, and fusion end temperature of the fiber-like cement reinforcement material based on DSC measurement were low values of 161°C, 166°C, 173°C and 180°C, respectively, and good shape retention after autoclave curing at 180°C was absent.

[0051]

#### Comparative Example 6

In Comparative Example 5, when the heat-treatment temperature of 170°C was used, fusion of the fiber took place during the course of the heat treatment and production of a fiber-like cement reinforcement material was not possible.

[0052]

		①			②	④ Table 1		⑤					⑥
		糸の物理的性質			延伸率 (%)	延伸糸の 拘束下融 解ピーク 温度 (℃)	拘束緊張 下の熱処 理温度 (℃)	耐熱性が57°比の繊維の物性					オートク レーブ養生 後の繊維 形状保持性
		IPF (%)	NFR (g/10 min)	Hv/Mn				融解ピーク 立ち上がり 温度 (℃)	融解ピーク 温度 (℃)	融解終了 温度 (℃)	熱収縮率 @170℃ (%)	熱収縮率 @175℃ (%)	
⑪	実施例 1	97.2	1.5	4.5	3.7	201	180	175	179	184	0	7.7	○
	実施例 2	97.2	1.5	4.5	3.7	201	183	177	182	187	0	6.4	○
	実施例 3	98.8	0.5	4.7	3.2	202	180	176	181	185	0	7.2	○
	実施例 4	97	10.0	3.7	4.5	183	180	174	179.5	185	0	9.3	○
⑫	比較例 1	97.2	1.5	4.5	3.7	201	163	158	165	173	77	融解	×
	比較例 2	97.2	1.5	4.5	3.7	201	165	167	169	177	65	融解	×
	比較例 3	94.2	2.0	5.2	4.0	170	155	158	163	168	融解	融解	×
	比較例 4	94.2	2.0	5.2	4.0	170	170	—	—	—	—	—	—
	比較例 5	96.7	40.0	4.2	5.0	172	160	161	166	173	融解	融解	×
	比較例 6	96.7	40.0	4.2	5.0	172	170	—	—	—	—	—	—

- Key:
- 1 Homopolypropylene resin
  - 2 Drawing ratio (times)
  - 3 Fusion peak temperature of drawn yarn under restraint state (°C)
  - 4 Heat-treatment temperature under restraint tension (°C)
  - 5 Properties of heat-resisting polypropylene fiber
  - 6 Shape retention of fiber after curing in autoclave

- 7 Fusion peak start-up temperature (°C)
- 8 Fusion peak temperature (°C)
- 9 Fusion end temperature (°C)
- 10 Heat shrinkage factor @ \_ (%)
- 11 Application Example \_
- 12 Comparative Example \_
- 13 None
- 14 Fusion

[0053]

#### Effect of the invention

In the polypropylene fiber or yarn with high heat resistance of the present invention, a homopolypropylene resin with high stereoregularity and specific flow properties is used and a heat treatment is provided at a high temperature after drawing under restraint tension; thus, the heat shrinkage factor at high temperature is low and a polypropylene fiber or yarn with high heat resistance with high melting point is produced, and when used as a cement reinforcement fiber, good shape retention can be achieved even under harsh curing conditions, and it can be used effectively as a reinforcement material.

#### Brief description of the figures

Figure 1 is the DSC measurement chart of the drawn yarns produced in Application examples 1, 2 and 4 under a restraint state.

Figure 2 is the DSC measurement chart of fibers produced in the application examples and comparative examples after heat treatment.

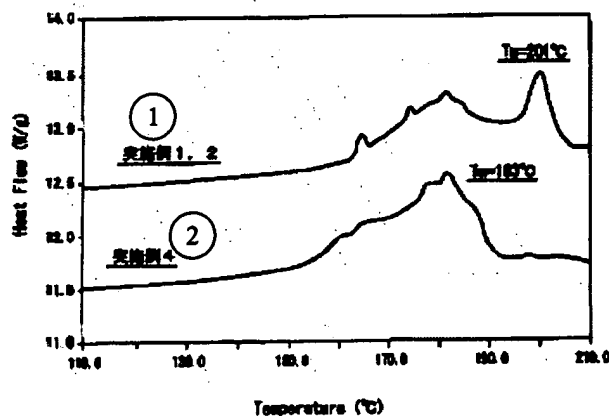


Figure 1

- Key: 1 Application Examples 1 and 2  
2 Application Example 4

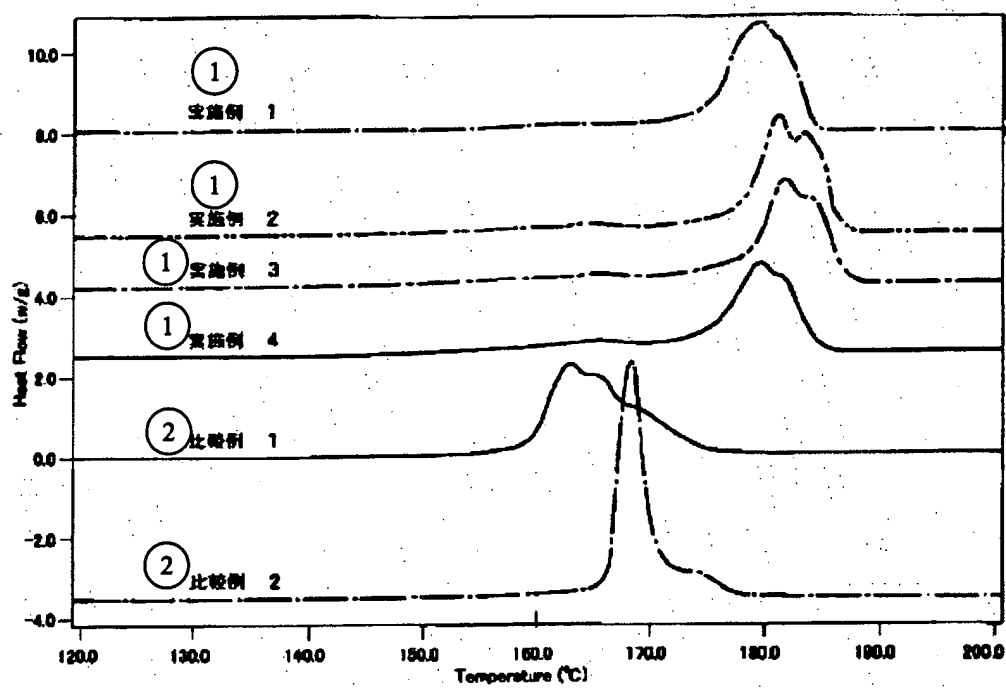


Figure 2

Key: 1 Application Example \_\_  
 2 Comparative Example \_\_